Interim Procedure for Verification of Emission Reductions for Alternative Diesel Fuels

(1) Purpose of Procedure for Verification of Emission Reductions for Alternative Diesel Fuels.

The purpose of the procedure is to provide the process for obtaining verification of emission reductions of particulates (PM) and oxides of nitrogen (NO_x) for alternative diesel fuels. For the purpose of this procedure, alternative diesel fuels mean fuels that are used in diesel engines that are not reformulated diesel fuels as defined in section 2281 and 2282 of Title 13, of the California Code of Regulations and do not require engine or fuel system modifications to operate on such fuels.

(2) Application for and Approval of Alternative Fuel Test Protocol.

The executive officer or designee, upon application of any producer or importer, may certify the emission reductions of PM and NO_x for alternative diesel fuels in accordance with the procedure. The applicant shall initially submit a proposed test protocol to the executive officer or designee. The proposed test protocol shall include: criteria pollutant and toxic emissions sampling and analysis consistent with the requirements of the procedure; test data showing that the candidate alternative diesel fuel parameters as shown in section (4); test data showing that the fuel to be used as the reference fuel meets the specifications identified in section (5); the identity of the entity proposed to conduct the tests described in section (6); and reasonably adequate quality assurance and quality control procedures; and notification of any outlier identification and exclusion procedure that will be used, and a demonstration that any procedure meets generally accepted statistical principles.

Within 20 days of receipt of a proposed test protocol, the executive officer or designee shall advise the applicant in writing either that it is complete or that specified additional information is required to make it complete. Within 15 days of submittal of additional information, the executive officer or designee shall advise the applicant in writing either that the information submitted makes the proposed test protocol complete or that specified additional information is still required to make it complete. Within 20 days after the proposed test protocol is deemed complete, the executive officer or designee shall either approve the changes necessary to make the test protocol consistent with the procedure or advise the applicant in writing of the changes necessary to make the test protocol consistent with the procedure. Any notification of approval of the test protocol shall include the name, telephone number, and address of the executive officer's designee to receive notifications pursuant to subsection (6)(F)(ii)**.** The tests shall not be conducted until the protocol is approved by the executive officer or designee.

(3) Application for and Approval of Alternative Fuel Emission Reduction Verification.

Upon completion of the tests, the applicant may submit an application for verification to the executive officer or designee. The application shall include the approved test protocol, all of the test data, copy of the complete test log prepared in accordance with subsection (6)(F)(ii), a demonstration that the candidate alternative diesel fuel meets the requirements for verification set forth in the procedure, and such other information as the executive officer or designee may reasonably require.

Within 20 days of receipt of an application, the executive officer or designee shall advise the applicant in writing either that it is complete or that specified additional information is required to make it complete. Within 15 days of submittal of additional information, the executive officer or designee shall advise the applicant in writing either that the information submitted makes the application complete or that specified additional information is still required to make it complete. Within 30 days after application is deemed complete, the executive officer or designee shall grant or deny the application. Any denial shall be accompanied by a written statement of the reasons for denial.

(4) Applicability, Description, and Fuel Parameters of Alternative Diesel Fuels.

The applicant shall describe the applicability of the alternative diesel fuel to diesel engines and identify any requirements for engine or fuel system modifications. The applicant shall provide a general description of the alternative diesel fuel that includes the fuel type, fuel characteristics, fuel properties, fuel formulation, and chemical composition. The applicant for the candidate alternative diesel fuel shall specify the following:

(a) Identity, chemical composition, and concentration of fuel additives
(b) Sulfur content
(c) Total aromatic content
(d) Total polycyclic aromatic hydrocarbon content
(e) Nitrogen content
(f) API gravity (density)
(g) Distillation temperature distribution information, initial boiling point (IBP), 10% recovered (REC), 5O% REC, 90% REC, and end point (EP)

The applicant shall provide information on the candidate alternative diesel fuel that may affect engine performance, engine wear, and safety. The applicant for the candidate alternative diesel fuel shall specify the following:

(a) Viscosity (engine performance)
(b) Fuel volatility (engine performance)
(c) Ignition quality (engine performance)
(d) Fuel operating temperatures (engine performance)
(e) Engine wear tendencies (engine wear)
(f) Corrosion (engine wear)
(g) Lubricity (engine wear)
(h) Fuel flash point (safety)

The applicant shall provide information on the candidate alternative diesel fuel to determine if there are chemicals in the fuel that may increase levels of toxic compounds or potentially form toxic compounds in the fuel. The applicant will conduct an analysis for metals and elements by a method specified by the applicant. Copper, iron, cerium, lead, cadmium, chromium, and phosphorus must be included in the analysis. Additional analysis for other toxic compounds may be required after reviewing the chemical composition of the candidate alternative diesel fuel and its additives. (Note: For emulsified diesel fuels, a toxic analysis of the diesel base fuel is not necessary.)

Upon approval of the executive officer or designee, an applicant may also specify different fuel parameters and test methods that are appropriate to better characterize the candidate alternative diesel fuel.

Upon review of the test protocol, the executive officer or designee may require additional fuel components, parameters, and specifications to be determined.

Example: Appropriate fuel parameters specified for emulsified diesel fuel.

(A) **Determination of fuel parameters for emulsified diesel fuel**. Emulsified diesel fuel is an alternative diesel fuel that is composed of water, diesel, and emulsified diesel fuel additives. The candidate base fuel (without water and emulsified diesel fuel additives) parameters and test methods are given in subsection (A)(i) and the candidate emulsified diesel fuel (with water and emulsified diesel fuel additives) parameters, chemical composition, and test methods are given in subsection (A)(ii). The applicant shall supply the candidate base diesel fuel used to make the

emulsified diesel fuel that will be used in the comparative testing pursuant in section (6). The following characteristics of the candidate base fuel and the candidate emulsified diesel fuel as determined by referenced test methods shall be the average of three tests.

(i) Candidate base fuel parameters. The candidate base fuel (without water and emulsified diesel fuel additives) shall meet the specification for 1-D or 2-D diesel fuel set forth in ASTM D975-81, which is incorporated herein by

reference. The candidate base fuel must be a commercially available certified diesel fuel that is in production in California. The applicant shall provide the following candidate base fuel parameters (the listed ASTM methods are incorporated herein by reference):

(a) Sulfur content (not to exceed 500 ppm) by ASTM D5453-93;

(b) Total aromatic hydrocarbon content, by ASTM D5186-96;

(c) Polycyclic aromatic hydrocarbon content, by ASTM D5186-96;

(d) Nitrogen content, by ASTM D4629-96;

(e) Cetane number, by ASTM D613-84;

(f) API Gravity (density) by ASTM 287-82;

(g) Distillation temperature distribution information, IBP, 10% REC, 50% REC, 90% REC, and EP by ASTM D86-96;

The applicant may also specify other parameters for the candidate base fuel, along with the test methods for determining the parameters.

(ii) Candidate emulsified diesel fuel parameters. The applicant shall supply the candidate emulsified diesel fuel (with water and emulsified diesel fuel additives) to be used in the comparative criteria pollutant testing and toxic emissions sampling and analysis pursuant to section (6). The applicant shall provide the following:

(a) The applicant shall specify the identity, chemical composition, and concentration of each additive to make the candidate emulsified diesel fuel, by a test method specified by the applicant and determined by the executive order to adequately determine the presence and concentration of the additive**.**

(b) The applicant shall specify the purity and quality of the water by a test method specified by the applicant and determined by the executive order to adequately characterized the quality and purity of the water. The applicant shall specify the presence and concentration of water by a test method specified by the applicant and determined by the executive order to adequately determine the presence and concentration of the water.

(c) Sulfur content (not to exceed 500 ppm) by ASTM D5453-93.

(d) Total aromatic hydrocarbon content by summing the aromatic content in the candidate base fuel as determined by ASTM D5186-96 (subsection(4)(A)(i)) and the aromatic content in the emulsified diesel fuel additives as determined by the applicant's method (subsection (4)(A)(ii)).

(e) Polycyclic aromatic hydrocarbon content by summing the polycyclic aromatic hydrocarbon content in the candidate base fuel as determined by ASTM D5186-96 (subsection(4)(A)(i)) and the polycyclic aromatic hydrocarbon content in the emulsified diesel fuel additives as determined by the applicant's method (subsection (4)(A)(ii)).

(f) Nitrogen content, by ASTM D4629-96;

(g) Cetane number, as determined by the applicant's method;

(h) Cold filter plugging, by ASTM D6371-99;

(i) Ash, by ASTM D482-95;

(j) Flash Point, by ASTM D93-80;

(k) API Gravity (density), as determined by the applicant's method;

(l) Distillation temperature distribution information, IBP, 10% REC, 50% REC, 90% REC, and EP (not applicable to emulsified diesel fuels)

The applicant may also specify other parameters for the additives to the candidate emulsified diesel fuel along with the test methods for determining the parameters.

(B) **Determination of additional fuel parameters in emulsified diesel fuel.**

The applicant shall provide information on the candidate emulsified diesel fuel (excluding the diesel base fuel) to determine if there are chemicals in the fuel that may increase levels of toxic compounds or potentially form toxic compounds in the fuel. The applicant will conduct an analysis for metals and elements by a method specified by the applicant. Copper, iron, cerium, lead, cadmium, chromium, and phosphorus must be included in the analysis. Additional analysis for other toxic compounds may be required after reviewing the chemical composition of the candidate emulsified diesel fuel and its component parts.

(5) Reference Fuel Specifications.

The reference fuel used in the comparative testing described in section (6) shall be produced from straight-run California diesel fuel by a hydrodearomatization process and shall have the characteristics set forth below under "Reference Fuel Specifications" (the listed ASTM methods are incorporated herein by reference):

Reference Fuel Specifications

Property	General Reference Fuel Specifications	ASTM Test Method
Sulfur Content	500 ppm max	D5453-93
Aromatic Hydrocarbon content, Vol. %	10% max	D5186-96
Polycyclic Aromatic Hydrocarbon content %	1.4% max	D5186-96
Nitrogen Content	10 ppm max	D4629-96
Natural Cetane Number	48 min	D613-84
Gravity, API	33-39	D287-82
Viscosity at 40^{o}, cSt	2.0-4.1	D445-83
Flash point, ^{o}F	130	D93-80
Distillation, ^{o}F IBP 10%REC 50%REC 90%REC EP	 340-420 400-490 470-560 550-610 580-660	D86-96

(6) Emissions Test Procedures for Particulates, Nitrogen Oxides, Soluble Organic Fraction, Hydrocarbons, and Toxics.

(A) **Criteria pollutants test requirements.** In each test of a fuel, exhaust emissions of NO_x, PM, and hydrocarbons shall be measured. In addition, for each test the soluble organic fraction (SOF) of the particulate matter in the exhaust emissions shall be determined in accordance with the Air Resources Board's "Test Method for Soluble Organic Fraction (SOF) Extraction " dated April 1989, which is incorporated herein by reference.

(B) **Toxic emissions sampling and analysis requirements.** Exhaust emissions of formaldehyde, acetaldehyde, benzene, toluene, ethyl benzene, xylenes, butadiene, and polycyclic aromatic hydrocarbons shall be sampled and analyzed as specified in Table 1.

Table 1. Toxics sampling and analysis [1,2]

Toxics	Method
Formaldehyde and acetaldehyde	ARB SOP 104
Benzene toluene, ethyl benzene, xylenes, and butadiene	ARB SOP 102/103
Polycyclic aromatic hydrocarbons	ARB method 429[3]

[1]Additional toxics sampling may be required depending on the chemical composition of the additives in the fuel.

[2]At a minimum tunnel blanks shall be required prior to and after conducting toxic emissions sampling for the reference fuel and candidate alternative diesel fuel.

[3]PAH sampling consists of a filter to collect particulate PAHs and XAD resin to collect volatile PAHs. The sampling protocol needs to be included in the test protocol. Analysis of the samples will be performed by ARB method 429.

(C) **Quality assurance.** Appropriate quality assurance procedures shall be required for all criteria pollutants and toxic emissions sampling and analysis.

(D) **Engine selection for emissions test program.** Exhaust emissions tests using the candidate alternative diesel fuel and the reference fuel shall be conducted in accordance with the "California Exhaust Emission Standards and Test Procedures for 1985 and Subsequent Model Heavy-Duty Diesel Powered Engines and Vehicles," as incorporated by reference in Title 13, California Code of Regulations, Section 1956.8(b). The tests shall be performed using a Detroit Diesel Corporation Series 60 engine or, if the executive officer or designee determines that the Series-60 is no longer representative of the post-1990 model year heavy-duty diesel engine fleet, another engine found by the executive officer or designee to be representative of such engines.

(E) **Selection of test facility.** The comparative emissions test program shall be conducted by a party or parties that are mutually agreed upon by the executive officer or designee and the applicant. The applicant shall be responsible for all costs of the comparative testing.

(F) **Test sequence for emissions test program**.

(i) The applicant shall use one of the following test sequences:

(a) If both cold start and hot start exhaust emission tests are conducted, a minimum of five exhaust emission tests shall be performed on the engine with each fuel, using either of the following sequences, where "R" is the reference fuel and "C" is the candidate alternative diesel fuel: RC CR RC CR RC (and continuing in the same order).

The engine mapping procedures and a conditioning transient cycle shall be conducted with the reference fuel before each cold start procedure using the reference fuel. The reference cycle used for the candidate alternative diesel fuel shall be the same as determined for the reference fuel.

(b) If only hot start exhaust emission tests are conducted, one of the following test sequences shall be used throughout the testing, where "R" is the reference fuel and "C" is the candidate alternative diesel fuel:

Alternative 1: RC CR RC CR (continuing in the same order for a given calendar day; a minimum of twenty individual exhaust emission tests must be completed with each fuel)

Alternative 2: RR CC RR CC (continuing in the same order for a given calendar day; a minimum of twenty individual exhaust emission tests must be completed with each fuel)

Alternative 3: RRR CCC RRR CCC (continuing in the same order for a given calendar day: a minimum of twenty-one individual exhaust emission tests must be completed with each fuel)

For all alternatives, an equal number of tests shall be conducted using the reference fuel and the candidate alternative diesel fuel on any given calendar day. At the beginning of each calendar day, the sequence of testing shall begin with the fuel that was tested at the end of the preceding day. The engine mapping procedures and a conditioning transient cycle shall be conducted at the beginning of each day for the reference fuel. The reference cycle used for the candidate alternative diesel fuel shall be the same as determined for the reference fuel. For alternatives 2 and 3, each paired or triplicate series of individual tests shall be averaged to obtain a single value, which would be used in the calculations conducted pursuant to subsection (7)(D).

(ii) The applicant shall submit a test schedule to the executive officer or designee at least one week prior to commencement of the tests. The test schedule shall identify the days on which the tests will be conducted, and shall provide for conducting test consecutively without substantial interruptions other than those resulting from the normal hours of operations at the test facility. The executive officer or designee shall be permitted to observe any tests. The party conducting the tests shall maintain a test log which identifies all tests conducted, all engine mapping procedures, all physical modifications to or operational tests of the engine, all recalibrations or other changes to the test instruments, and all interruptions between tests, and the reason for each interruption. The party conducting the tests or the applicant shall notify the

executive officer or designee by telephone and in writing of any unscheduled interruption resulting in a test delay of 48 hours or more, and the reason for such delay. Prior to restarting the test, the applicant or person conducting the tests shall provide the executive officer or designee with a revised schedule for the remaining tests. All tests conducted in accordance with the test schedule, other than any test rejected in accordance with an outlier identification and exclusion procedure included in the approved test protocol, shall be included in the comparison of emissions pursuant to section (7).

(iii) Upon approval of the executive officer or designee, the applicant may specify an alternative test sequence to subsection (6)(F)(i). The applicant shall provide the rationale demonstrating that the alternative test sequence better characterizes the average emissions difference between the reference fuel and the alternative diesel fuel.

(7) Procedure for Evaluating Emissions Test Data.

The average emissions of PM and NO_x, during testing with the candidate alternative diesel fuel shall be compared to the average emissions during testing with reference fuel, applying one-sided Student's t statistics as set forth in Snedecar and Cochran, Statistical Methods (7th ed.), page 91, Iowa State University Press, 1980, which is incorporated herein by reference. Also, the average emissions of hydrocarbons and toxic emissions during testing with the candidate alternative diesel fuel shall be compared to the average emissions during testing with the reference fuel. The executive officer or designee shall issue a verification pursuant to this paragraph only if he or she makes all of the determinations set forth in subsections (7)(A), (7)(B), (7)(C), (7)(E), and (7)(F).

(A) **NO_x reduction specification.** The average individual emissions of NO_x during testing with the candidate alternative diesel fuel shall be specified as a percent reduction of the average individual emissions of NO_x**,** during testing with the reference fuel.

(B) **PM reduction specification**. The average individual emissions of PM during testing with the candidate alternative diesel fuel shall be specified as a percent reduction of the average emissions of PM during testing with reference fuel.

(C) **SOF specification**. The average individual emissions of SOF during testing of the candidate alternative diesel fuel shall not result in a net increase in the average individual emissions of SOF during testing of the reference diesel fuel.

(D) **Calculation of PM, NO_x, and SOF emissions.** In order for the determinations in subparagraphs (A), (B), and (C) to be made, for each referenced pollutant the candidate alternative diesel fuel shall satisfy the following relationship:

Equation for PM determination

$X_c = fX_r + \delta - S_p \cdot sqrot\ 2/n \cdot t\ (a, 2n-2)$

Where: X_c = Average PM emissions during testing with the candidate alternative diesel fuel

f = Emissions of the candidate fuel expressed as a fraction of the emissions of the reference fuel

X_r = Average PM emissions during testing with reference fuel

δ = tolerance level equal to actual $S_p \cdot sqrot\ 2/n \cdot t\ (a, 2n-2)$ of the reference fuel but not more than 4 percent of X_r for PM.

S_p = Pooled standard deviation

t(a, 2n-2) = The one-sided upper percentage point of t distribution with a = 0.15 and 2n-2 degrees of freedom

n = Number of tests of candidate and reference fuel

Equation for NO_x determination.

$X_c = fX_r + \delta - S_p \cdot sqrot\ 2/n \cdot t\ (a, 2n-2)$

Where: X_c = Average NO_x emissions during testing with the candidate alternative diesel fuel

X_r = Average NO_x emissions during testing with reference fuel

f = Emissions of the candidate fuel expressed as a fraction of the emissions of the reference fuel

δ = tolerance level equal to actual $S_p \cdot sqrot\ 2/n \cdot t\ (a, 2n-2)$ of the reference fuel but not more than 2 percent of X_r for NO_x,

S_p = Pooled standard deviation for the reference fuel

t(a, 2n-2) = The one-sided upper percentage point of t distribution with a = 0.15 and 2n-2 degrees of freedom

n = Number of tests of candidate and reference fuel

Equation for SOF determination.

$X_c < X_r + \delta - S_p \cdot \text{sqrot } 2/n \cdot t\ (a, 2n-2)$

Where: X_c = Average SOF emissions during testing with the candidate alternative diesel fuel

X_r = Average SOF emissions during testing with reference fuel

δ = tolerance level equal to actual $S_p \cdot \text{sqrot } 2/n \cdot t\ (a, 2n-2)$ of the reference fuel but not more than 12 percent of X_r for SOF,

S_p = Pooled standard deviation

t(a, 2n-2) = The one-sided upper percentage point of t distribution with a = 0.15 and 2n-2 degrees of freedom

n = Number of tests of candidate and reference fuel

(E) **Hydrocarbon emissions specification.** The average individual emissions of hydrocarbons during testing of the candidate alternative diesel fuel shall be at least 25% lower than any applicable diesel exhaust vehicle standard for emissions of hydrocarbons.

(F) **Toxic emissions specification**. The sum of average individual emissions of toxics during testing of the candidate alternative diesel fuel shall not result in a net increase in overall toxicity over the sum of average individual emissions of toxics during testing of the reference diesel fuel.

(8) Determine the Effect the Candidate Alternative Diesel Fuel has on Engine Performance and Engine Wear/Damage.

The applicant shall provide the ARB executive officer or designee test data showing that the candidate alternative diesel fuel does not adversely affect the performance and operation of diesel engines or cause premature wear or cause damage to diesel engines. This shall include but not limited to lubricity, corrosion, and damage to engine parts such as fuel injector tips. The applicant shall provide data showing under what temperature and conditions does the candidate alternative diesel fuel remains stable and usable in California.

(9) The candidate fuel shall be in compliance with applicable federal, state, and local government requirements.

Two agencies that need to be contacted when marketing fuel in California are the US EPA and the California Dept. of Food and Agriculture. Contacts are listed below.

James Caldwell
Office of Transport of Air Quality
USEPA Head Quarters
Ariel Rios Blvd.
1200 Pennsylvania Ave, N.W.
Washington DC 20468
Ph (202) 564-9303

David Lazier
Pertrolem Products/Weighmaster Enforcement Brach
Division of Measurement Standards
Dept. of Food and Agriculture
8500 Fruitridge Road, Sacramento CA 95826
Ph (916) 229-3000

Additional government agencies such as the California Energy Commission, Area Council Governments, and Local Air Quality Management Districts may be contacted to facilitate the marketing of alternative diesel fuel in California.